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# मानक

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IS 12308-5 (1991): Methods for Chemical Analysis of Cast Iron and Pig Iron, Part 5: Determination of phosphorus by alkalimetric method (for phosphorus 0.01 to 0.50 percent) [MTD 6: Pig iron and Cast Iron]



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भारतीय मानक  
ढलवां लोहे एवं कच्चे लोहे के रासायनिक विश्लेषण  
की पद्धतियाँ

भाग 5 क्षारमितीय पद्धति से फॉस्फोरस ( 0.01 से 0.50 प्रतिशत तक ) ज्ञात करना

*Indian Standard*

METHODS OF CHEMICAL ANALYSIS OF CAST  
IRON AND PIG IRON

PART 5 DETERMINATION OF PHOSPHORUS ( 0.01 TO 0.50 PERCENT )  
BY ALKALIMETRIC METHOD

UDC 669.162.275.12 + 669.13 : 543.241 [ 543.847 ]

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BUREAU OF INDIAN STANDARDS  
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## FOREWORD

This Indian Standard ( Part 5 ) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

Chemical analysis of cast iron and pig iron was covered in IS 228 : 1959 'Methods of chemical analysis of pig iron, cast iron and plain carbon and low alloy steels ( *revised* )'. During its second revision it was decided that a comprehensive series should be prepared for chemical analysis of all types of steels and the other covering the chemical analysis of cast iron and pig iron. Accordingly IS 228 on revision was published in several parts covering chemical analysis of various steels only and a separate series of standards under IS 12308 is being published for chemical analysis of cast iron and pig iron. This standard ( Part 5 ) is one in the latter series. The other parts in the series are:

IS 12308 Methods for chemical analysis of cast iron and pig iron:

- Part 1 Determination of total carbon by thermal conductivity method
- Part 2 Determination of sulphur by iodimetric titration method
- Part 3 Determination of manganese by periodate spectrophotometric method
- Part 4 Determination of total carbon, graphitic carbon and combined carbon by gravimetric method
- Part 6 Determination of silicon ( for silicon 0.1 to 6.0 percent )
- Part 7 Determination of nickel by dimethylglyoxime ( Gravimetric ) method ( for nickel 0.5 to 36 percent )
- Part 8 Determination of chromium by persulphate oxidation method ( for chromium 0.1 to 28 percent )
- Part 9 Determination of molybdenum by thiocyanate ( Spectrophotometric ) method ( for molybdenum 0.1 to 1.0 percent )
- Part 10 Determination of manganese ( up to 7.0 percent ) by arsenite ( Volumetric ) method
- Part 11 Determination of total carbon by the direct combustion volumetric method ( for carbon 1.50 to 4.50 percent )

The method given in Part 5 for determination of phosphorus has been updated on the basis of experience gained over the period.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values ( *revised* )'.

## Indian Standard

# METHODS OF CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

### PART 5 DETERMINATION OF PHOSPHORUS ( 0.01 TO 0.50 PERCENT ) BY ALKALIMETRIC METHOD

#### 1 SCOPE

This standard ( Part 5 ) prescribes the method for determination of phosphorus, in the range of 0.01 to 0.50 percent, in cast iron and pig iron.

#### 2 REFERENCE

The Indian Standard IS 264 : 1976 'Nitric acid ( *second revision* )' is necessary adjunct to this standard.

#### 3 SAMPLING

The sample shall be drawn and prepared as prescribed in the relevant Indian Standard.

#### 4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water shall be employed in the test.

#### 5 METHOD

##### 5.1 Outline of the Method

Phosphorus is converted to orthophosphoric acid and precipitated as ammonium phosphomolybdate. The precipitate is dissolved in known excess of standard sodium hydroxide solution and excess is titrated against standard nitric acid solution.

##### 5.2 Reagents

**5.2.1 Concentrated Nitric Acid** — rd = 1.42 ( conforming to IS 264 : 1976 ).

**5.2.2 Dilute Nitric Acid** — 1 percent, 2 percent and 2 : 3 ( v/v ).

**5.2.3 Potassium Permanganate Solution** — 2 percent ( m/v ).

**5.2.4 Sodium Nitrite** — 2 percent ( m/v ).

**5.2.5 Concentrated Ammonium Hydroxide** — rd = 0.90.

##### 5.2.6 Ammonium Molybdate Solution

Add *Solution A* ( see 5.2.6.1 ) slowly and with constant stirring to *Solution B* ( see 5.2.6.2 ) kept cool in a cold waterbath. Add 10 ml of ammonium phosphate solution ( 1 g/l ) and keep the solution at least for 24 hours. Filter the

solution through medium textured filter paper before use.

##### 5.2.6.1 Solution A

Dissolve 100 g of molybdic acid (  $\text{MoO}_3$ , 85 percent ), or 118 g of ammonium molybdate in a mixture of 145 ml of ammonium hydroxide ( rd = 0.90 ) and 270 ml of water. Cool the solution.

##### 5.2.6.2 Solution B

Add 300 ml of concentrated nitric acid to 700 ml of water and cool.

**5.2.7 Potassium Nitrate Solution** — 1 percent ( m/v ).

**5.2.8 Phenolphthalein Indicator Solution** — 0.2 percent.

Dissolve 0.2 g of phenolphthalein powder in 80 ml of rectified spirit and make up to 100 ml with the rectified spirit.

**5.2.9 Standard Sodium Hydroxide Solution** — 0.1 N.

Dissolve 4.5 g of sodium hydroxide in one litre of freshly boiled and cooled distilled water, and standardize against standard nitric acid ( 5.2.10 ).

**5.2.10 Standard Nitric Acid Solution** — 0.1 N.

Dilute 7 ml of concentrated nitric acid to one litre with freshly boiled distilled water. Standardize against sodium carbonate previously heated at 275-300°C and cooled. 1 ml of this solution will then be equivalent to 0.000 135 g of phosphorus.

##### 5.3 Procedure

**5.3.1** Transfer 0.200 to 2.000 g of sample ( containing 0.2 to 2 mg of P ) to 250-ml beaker and dissolve in 30 ml of dilute nitric acid ( 2 : 3 ) ( see Notes 1 and 2 ). Digest until dissolve. Evaporate to syrupy consistency and cool. Add 2 ml of concentrated nitric acid and 50 ml of water. Filter and wash 4 to 5 times with dilute nitric acid ( 1 percent ). Take the clear filtrate and while boiling add potassium permanganate solution dropwise till a brown precipitate of manganese dioxide appears. Boil for another 2 minutes. Add sodium nitrite solution dropwise to clear the brown precipitate of manganese. Add another 2 to 3 drops of sodium nitrite and boil for 5 minutes to expel oxides of nitrogen.

Cool the solution and to the cooled solution add ammonium hydroxide drop by drop till neutralization. Add 2 to 3 drops of nitric acid to clear the solution. Add 5 ml of nitric acid. The volume at this stage should be about 80 ml. Heat the solution to 60-80°C and transfer to a 250-ml conical flask.

#### NOTES

1 If sample is not soluble in nitric acid, dissolve in mixture of nitric acid and hydrochloric acid. When sample is decomposed add perchloric acid and evaporate nearly to dryness. Add 30 ml of nitric acid and proceed.

2 If vanadium is present, add 2 to 3 ml of sodium nitrite during the removal of manganese dioxide and precipitation of phosphate is performed at 10 to 20°C.

**5.3.2** To the warm solution add 30 ml of ammonium molybdate solution. Stopper the flask, shake vigorously for a few minutes and allow to stand for half an hour. Filter the precipitate through medium textured filter paper or filter paper pulp. Wash the precipitate with dilute nitric acid solution ( 1 percent ), once or twice. Finally wash the flask, the precipitate and the paper, with 5 ml portions of potassium nitrate wash solution until 10 ml of the filtrate collected in a test tube does not consume more than one drop of the standard sodium hydroxide solution ( 5.2.9 ) in presence of a drop of phenolphthalein indicator solution.

NOTE — Generally 3 or 4 washings with potassium

nitrate solution are sufficient for the precipitate to be acid free.

**5.3.3** Transfer the paper and precipitate to the flask in which precipitation was carried out. Add about 25 ml of water, and a known volume of standard sodium hydroxide solution ( which should be 2 to 5 ml in excess ) and shake to dissolve the precipitate. Dilute to about 100 ml and titrate with standard nitric acid solution using phenolphthalein as indicator.

**5.3.4** Carry out a blank determination following the same procedure as specified in 5.3.1 to 5.3.3 and using the same quantity of all reagents.

#### 5.4 Calculation

Phosphorus content,  
percent by mass 
$$= \frac{(B - A) \times C}{D} \times 100$$

where

$B$  = volume, in ml, of standard nitric acid solution required for the blank ( 7.1 );

$A$  = volume, in ml, of standard nitric acid solution required for the sample ( see 5.3.3 );

$C$  = phosphorus equivalent of 1 ml standard nitric acid solution ( see 5.2.10 ); and

$D$  = mass, in g, of the sample taken for test.

**5.5 Reproducibility** —  $\pm 0.001$  5 percent.

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